Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Aqua[3-carboxy-2-(3,5-dibromo-2-oxidobenzylamino-κ^2N,O)propanoato-κO^1](dimethylformamide-κO)copper(II) dimethylformamide solvate monohydrate

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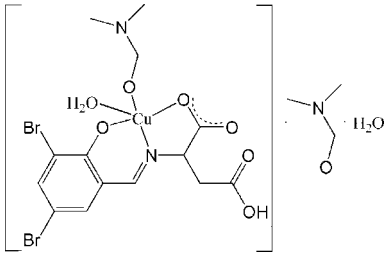
Received 4 April 2007; accepted 23 August 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.038; wR factor = 0.087; data-to-parameter ratio = 14.1.

In the title compound, $[Cu(C_{11}H_7Br_2NO_5)(C_3H_7NO)(H_2O)]\cdot C_3H_7NO\cdot H_2O$, the Cu^{II} atom is coordinated in a slightly distorted square-pyramidal geometry defined by two O atoms and one N atom from a 3-carboxy-2-(3,5-dibromo-2-oxidobenzylamino)propanoate ligand, one O atom from a dimethylformamide (DMF) molecule in the basal plane, and by one O atom from an aqua ligand in the apical position. In the crystal structure, the water molecules are linked by O—H···O hydrogen bonds into chains which resemble a pearl necklace. The molecules form a three-dimensional supramolecular network through O—H···O hydrogen bonds and C—H···Br hydrogen bonds.

Related literature

For related literature, see: Cheruzel *et al.* (2003); Feng *et al.* (2007); Ghosh & Bharadwaj (2003); Ghosh & Bharadwaj (2004); Ghosh *et al.* (2005); Moorthy *et al.* (2002); Neogi & Bharadwaj (2005); Raghuraman *et al.* (2003); Tajkhorshid *et al.* (2002); Ugalde *et al.* (2000); Ye *et al.* (2004); Zaslavsky & Gennis (2000); Zhang *et al.* (2005).



Experimental

Crystal data

$[Cu(C_{11}H_7Br_2NO_5)(C_3H_7NO)(H_2O)]\cdot C_3H_7NO\cdot H_2O$
M_r = 638.76
Monoclinic, $C2/c$
a = 46.176 (3) Å
b = 5.1633 (19) Å
c = 21.793 (2) Å
β = 116.186 (4)°
V = 4662.6 (18) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 4.42 mm^{-1}
T = 298 (2) K
0.56 × 0.10 × 0.07 mm

Data collection

Bruker SMART 1K CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.191, T_{max} = 0.747
11320 measured reflections
4091 independent reflections
3197 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.087
S = 1.04
4091 reflections
290 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O5	1.887 (3)	Cu1—O6	1.969 (3)
Cu1—N1	1.916 (3)	Cu1—O9	2.556 (3)
Cu1—O1	1.950 (3)		
O5—Cu1—N1	93.51 (12)	O1—Cu1—O6	90.42 (11)
O5—Cu1—O1	171.55 (12)	O5—Cu1—O9	99.55 (11)
N1—Cu1—O1	84.86 (12)	N1—Cu1—O9	85.39 (11)
O5—Cu1—O6	90.85 (11)	O1—Cu1—O9	88.60 (11)
N1—Cu1—O6	174.78 (12)	O6—Cu1—O9	96.73 (11)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O7^{i}	0.82	1.76	2.565 (5)	169
O8—H18···O2^{ii}	0.85	1.93	2.780 (4)	176
O8—H19···O8^{iii}	0.85	2.04	2.889 (3)	175
O9—H20···O8^{iv}	0.85	2.06	2.904 (4)	170
O9—H21···O1^{v}	0.85	2.28	3.116 (4)	167
C3—H3A···Br11^{vi}	0.97	3.13	3.605 (2)	112

Symmetry codes: (i) $x, -y+1, z-\frac{1}{2}$; (ii) $x, -y+1, z+\frac{1}{2}$; (iii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (iv) $-x+\frac{3}{2}, -y+\frac{1}{2}, -z+1$; (v) $x, y-1, z$; (vi) $x, -y, z-\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

The authors acknowledge financial support from the Key Laboratory of Nonferrous Metal Materials and Processing Technology, Ministry of Education, People's Republic of China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2034).

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supplementary materials

Acta Cryst. (2007). E**63**, m2631-m2632 [**doi:10.1107/S1600536807041517**]

Aqua[3-carboxy-2-(3,5-dibromo-2-oxidobenzylamino-κ^2N,O)propanoato-κO^1](dimethylformamide-κO)copper(II) dimethylformamide solvate monohydrate

L.-X. Jin, S.-H. Zhang, Z. Liu and G.-Z. Li

Comment

Rencently, compounds containing dimmer water (Ghosh & Bharadwaj, 2003), water ring (Moorthy *et al.*, 2002, Ugalde *et al.*, 2000, Ghosh & Bharadwaj, 2004), water chain (Neogi & Bharadwaj, 2005, Cheruzel *et al.*, 2003, Ghosh *et al.*, 2005), metal-water chain (Ye *et al.*, 2004), water network (Zhang *et al.*, 2005), water-methanol intermix clusters (Raghuraman *et al.*, 2003), and water ring chain (Feng, *et al.*, 2007) give rise to considerable interest. Because these dimmer water, water ring, water chain, metal-water chain, water network, water ring chain and intermix clusters are potentially important form of water that is poorly understood. Water chains appear to facilitate the selective permeation (Tajkhorshid *et al.*, 2002) of water across membranes and also to be important (Zaslavsky, & Gennis, 2000) in the control of proton fluxes in a variety of biomolecules.

The title compound, (I), is a Cu^{II} complex containing a ligand constructed from 2-(3,5-dibromo-2-hydroxy-benzylamino)-succinic acid and 3,5-dichloro-2-hydroxy-benzaldehyde. Each Cu^{II} atom is coordinated by two O atoms and one N atom from L^{2-} and one O atom from DMF and one O atom from H_2O to furnish a slightly distorted tetragonal pyramidal(Table 1). The asymmetric unit (Fig. 1) comprises one Cu^{II} complexes, one water molecule and one DMF molecule. The water molecules are linked by O—H···O hydrogen bonds (Table 2), forming a 'peral necklace' water chains. In the 'pearl necklace' water chains, the O8 water act as thread and the O9 water as pearl along 101 plan(Fig. 2). The water chains lie between layers of Cu^{II} complexes (Fig. 3), forming O—H···O hydrogen bonds to the O2 atoms of the carboxylate groups. The complex are further constructed three-dimensional supramolecular network through O—H···O hydrogen bond and C—H···Br hydrogen bond (Fig. 3).

Experimental

A solution of aspartic acid (0.059 g, 0.5 mmol) and pottasium hydroxide (0.028 g, 0.5 mmol) in distilled water (10 ml) was added slowly to a solution of 3,5-dibromo-2-hydroxybenzaldehyde (0.5 mmol, 0.140 g) in ethanol (10 ml). The mixture was stirred for 1 h at 323 K, then added slowly to a solution of copper(II) nitrate (0.121 g, 0.5 mmol) in distilled water (10 ml). This mixture was stirred and refluxed for 2 h at 323 K; The precipitate was separated by filtration. Dregs were dissolved in DMF. The solution was filtered and the filtrate was left to stand at room temperature. Blue strips suitable for X-ray diffraction were obtained in a yield of 65% (based on copper nitrate).

Refinement

H atoms of the water molecule were located in a difference Fourier map. The O—H distances were normalized to 0.85 Å and the H atoms were allowed to ride on the O atom, with U_{iso}(H) = 1.5 U_{eq}(O). All other H atoms were positioned

geometrically and refined as riding, with C—H distances of 0.93–0.98 Å and U_{iso}(H) = 1.2 U_{eq}(C) and with O—H distances of 0.82 Å and U_{iso}(H) = 1.5 U_{eq}(O).

Figures

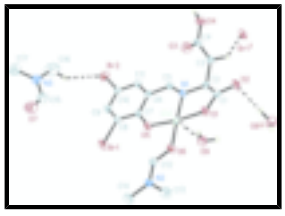

Fig. 1. The asymmetric unit of (I), showing displacement ellipsoids drawn at the 30% probability level for non-H atoms. The dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted.

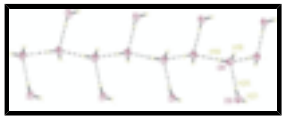

Fig. 2. Water molecules linked by O—H···O hydrogen bonds (dashed lines) that form chains. Displacement ellipsoids are shown at the 30% probability level for O atoms.

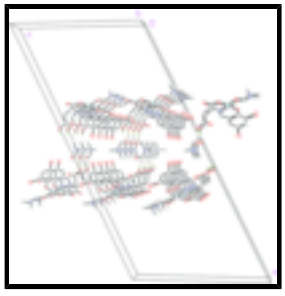

Fig. 3. Projection of (I) along b, showing layers of Cu^{II} complexes with the water molecules lying between them. Dashed lines denote hydrogen bonds and H atoms not involved in hydrogen bonding have been omitted.

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Crystal data

$[Cu(C_{11}H_7Br_2NO_5)(C_3H_7NO)(H_2O)]\cdot C_3H_7NO\cdot H_2O$	F_{000} = 2552
M_r = 638.76	D_x = 1.820 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 4147 reflections
a = 46.176 (3) Å	θ = 2.7–25.7°
b = 5.1633 (19) Å	μ = 4.42 mm^{-1}
c = 21.793 (2) Å	*T* = 298 (2) K
β = 116.186 (4)°	Strip, blue
V = 4662.6 (18) Å^3	0.56 × 0.10 × 0.07 mm
Z = 8	

Data collection

Bruker SMART 1K CCD diffractometer	4091 independent reflections
Radiation source: fine-focus sealed tube	3197 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.044

Detector resolution: 10 pixels mm^{-1}

T = 298(2) K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 2002)

T_{min} = 0.191, T_{max} = 0.747

11320 measured reflections

θ_{max} = 25.0º

θ_{min} = 1.9º

h = −54→33

k = −6→6

l = −25→25

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.038

$wR(F^2)$ = 0.087

S = 1.04

4091 reflections

290 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0358P)^2 + 6.521P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.002

$\Delta\rho_{max}$ = 0.47 e $Å^{-3}$

$\Delta\rho_{min}$ = −0.34 e $Å^{-3}$

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.667762 (11)	0.45742 (9)	0.32640 (2)	0.03304 (14)
Br1	0.631916 (12)	0.16100 (10)	0.48947 (2)	0.05071 (15)
Br2	0.550915 (11)	−0.56975 (9)	0.29641 (3)	0.05290 (15)
N1	0.64487 (7)	0.2519 (6)	0.24615 (15)	0.0299 (7)
N2	0.70705 (8)	0.8065 (7)	0.51375 (15)	0.0370 (8)
N3	0.49942 (10)	0.0691 (9)	0.3994 (2)	0.0650 (12)
O1	0.68006 (6)	0.6679 (5)	0.26737 (13)	0.0379 (7)
O2	0.67757 (7)	0.6785 (6)	0.16312 (13)	0.0459 (7)
O3	0.59772 (7)	0.6053 (6)	0.12787 (15)	0.0564 (9)
O4	0.58105 (8)	0.3707 (7)	0.03307 (14)	0.0592 (9)
H4	0.5656	0.4695	0.0201	0.089*

O5	0.64999 (6)	0.2798 (5)	0.37752 (12)	0.0377 (7)
O6	0.69090 (6)	0.6953 (5)	0.40330 (12)	0.0380 (7)
O7	0.52932 (10)	0.3581 (10)	0.4777 (2)	0.1011 (15)
O8	0.74314 (7)	0.2116 (6)	0.71684 (16)	0.0562 (8)
H18	0.7230	0.2380	0.7012	0.084*
H19	0.7466	0.0605	0.7341	0.084*
O9	0.71561 (7)	0.1607 (6)	0.34796 (14)	0.0518 (8)
H20	0.7256	0.1984	0.3244	0.078*
H21	0.7051	0.0219	0.3315	0.078*
C1	0.67179 (9)	0.5756 (8)	0.20757 (19)	0.0325 (9)
C2	0.65577 (9)	0.3065 (8)	0.19336 (18)	0.0338 (9)
H2	0.6732	0.1827	0.2015	0.041*
C3	0.63146 (9)	0.2660 (8)	0.11995 (18)	0.0366 (10)
H3A	0.6421	0.2968	0.0909	0.044*
H3B	0.6247	0.0861	0.1142	0.044*
C4	0.60197 (10)	0.4333 (8)	0.0956 (2)	0.0386 (10)
C5	0.62537 (9)	0.0658 (8)	0.23949 (19)	0.0322 (9)
H5	0.6167	−0.0235	0.1982	0.039*
C6	0.61567 (9)	−0.0170 (8)	0.29146 (19)	0.0314 (9)
C7	0.62860 (9)	0.0981 (7)	0.35723 (18)	0.0304 (9)
C8	0.61662 (9)	0.0012 (7)	0.40234 (19)	0.0323 (9)
C9	0.59465 (9)	−0.1957 (8)	0.3859 (2)	0.0371 (10)
H9	0.5880	−0.2573	0.4177	0.044*
C10	0.58250 (9)	−0.3019 (7)	0.3207 (2)	0.0353 (9)
C11	0.59296 (9)	−0.2178 (8)	0.2745 (2)	0.0345 (9)
H11	0.5850	−0.2942	0.2315	0.041*
C12	0.69048 (9)	0.6671 (8)	0.4600 (2)	0.0359 (9)
H12	0.6774	0.5373	0.4638	0.043*
C13	0.72672 (11)	1.0211 (9)	0.5119 (2)	0.0540 (12)
H13A	0.7238	1.0466	0.4659	0.081*
H13B	0.7490	0.9842	0.5410	0.081*
H13C	0.7204	1.1752	0.5276	0.081*
C14	0.70679 (12)	0.7506 (10)	0.5795 (2)	0.0557 (13)
H14A	0.6934	0.6023	0.5748	0.084*
H14B	0.6984	0.8973	0.5935	0.084*
H14C	0.7284	0.7156	0.6133	0.084*
C15	0.52653 (13)	0.1844 (12)	0.4396 (3)	0.0691 (15)
H15	0.5452	0.1250	0.4382	0.083*
C16	0.49831 (18)	−0.1326 (14)	0.3523 (4)	0.122 (3)
H16A	0.5198	−0.1692	0.3584	0.183*
H16B	0.4853	−0.0760	0.3062	0.183*
H16C	0.4891	−0.2863	0.3611	0.183*
C17	0.46963 (14)	0.1399 (15)	0.3995 (3)	0.097 (2)
H17A	0.4737	0.2536	0.4373	0.145*
H17B	0.4588	−0.0129	0.4038	0.145*
H17C	0.4563	0.2266	0.3574	0.145*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0410 (3)	0.0322 (3)	0.0282 (3)	−0.0060 (2)	0.0174 (2)	−0.0026 (2)
Br1	0.0727 (3)	0.0537 (3)	0.0363 (2)	−0.0067 (3)	0.0336 (2)	−0.0012 (2)
Br2	0.0474 (3)	0.0431 (3)	0.0706 (3)	−0.0103 (2)	0.0282 (2)	0.0033 (2)
N1	0.0345 (17)	0.0314 (18)	0.0297 (16)	0.0006 (16)	0.0197 (14)	−0.0012 (14)
N2	0.0403 (19)	0.040 (2)	0.0302 (17)	−0.0008 (17)	0.0149 (15)	−0.0066 (16)
N3	0.060 (3)	0.066 (3)	0.060 (3)	−0.004 (2)	0.018 (2)	−0.012 (2)
O1	0.0504 (16)	0.0331 (16)	0.0333 (15)	−0.0083 (14)	0.0213 (13)	−0.0022 (13)
O2	0.0609 (19)	0.0492 (19)	0.0368 (16)	−0.0100 (16)	0.0300 (15)	0.0027 (14)
O3	0.060 (2)	0.052 (2)	0.0504 (19)	0.0087 (17)	0.0179 (16)	−0.0108 (16)
O4	0.056 (2)	0.076 (3)	0.0342 (17)	0.0043 (18)	0.0098 (15)	−0.0064 (17)
O5	0.0487 (17)	0.0384 (17)	0.0305 (14)	−0.0143 (14)	0.0214 (12)	−0.0066 (13)
O6	0.0495 (17)	0.0360 (17)	0.0305 (15)	−0.0091 (14)	0.0194 (13)	−0.0041 (12)
O7	0.072 (3)	0.130 (4)	0.086 (3)	−0.023 (3)	0.021 (2)	−0.053 (3)
O8	0.0523 (19)	0.049 (2)	0.072 (2)	−0.0015 (16)	0.0317 (16)	0.0006 (17)
O9	0.0545 (18)	0.0473 (19)	0.0528 (19)	−0.0079 (16)	0.0228 (15)	0.0021 (16)
C1	0.031 (2)	0.034 (2)	0.032 (2)	0.0023 (18)	0.0140 (17)	−0.0036 (18)
C2	0.041 (2)	0.033 (2)	0.034 (2)	−0.0002 (19)	0.0231 (18)	−0.0036 (18)
C3	0.045 (2)	0.037 (2)	0.033 (2)	−0.005 (2)	0.0217 (18)	−0.0082 (19)
C4	0.049 (3)	0.037 (3)	0.033 (2)	−0.008 (2)	0.021 (2)	−0.0044 (19)
C5	0.035 (2)	0.034 (2)	0.027 (2)	0.0017 (19)	0.0142 (17)	−0.0027 (18)
C6	0.031 (2)	0.029 (2)	0.037 (2)	0.0018 (18)	0.0175 (17)	−0.0007 (17)
C7	0.036 (2)	0.029 (2)	0.030 (2)	0.0060 (19)	0.0171 (17)	0.0046 (17)
C8	0.038 (2)	0.028 (2)	0.033 (2)	0.0050 (18)	0.0171 (17)	0.0029 (17)
C9	0.041 (2)	0.035 (2)	0.042 (2)	0.005 (2)	0.0245 (19)	0.009 (2)
C10	0.033 (2)	0.028 (2)	0.044 (2)	−0.0021 (18)	0.0168 (19)	0.0005 (19)
C11	0.035 (2)	0.033 (2)	0.035 (2)	0.0025 (19)	0.0155 (18)	−0.0018 (18)
C12	0.036 (2)	0.034 (2)	0.038 (2)	−0.0040 (19)	0.0173 (18)	−0.0038 (19)
C13	0.065 (3)	0.044 (3)	0.053 (3)	−0.021 (3)	0.026 (2)	−0.013 (2)
C14	0.068 (3)	0.067 (3)	0.038 (2)	−0.005 (3)	0.028 (2)	−0.005 (2)
C15	0.058 (3)	0.077 (4)	0.062 (3)	0.003 (3)	0.017 (3)	−0.001 (3)
C16	0.110 (6)	0.089 (5)	0.135 (6)	0.014 (5)	0.024 (5)	−0.047 (5)
C17	0.069 (4)	0.134 (6)	0.090 (5)	−0.019 (4)	0.038 (4)	−0.025 (5)

Geometric parameters (Å, °)

Cu1—O5	1.887 (3)	C2—H2	0.9800
Cu1—N1	1.916 (3)	C3—C4	1.498 (6)
Cu1—O1	1.950 (3)	C3—H3A	0.9700
Cu1—O6	1.969 (3)	C3—H3B	0.9700
Cu1—O9	2.556 (3)	C5—C6	1.454 (5)
Br1—C8	1.898 (4)	C5—H5	0.9300
Br2—C10	1.907 (4)	C6—C11	1.403 (5)
N1—C5	1.281 (5)	C6—C7	1.417 (5)
N1—C2	1.472 (5)	C7—C8	1.415 (5)
N2—C12	1.299 (5)	C8—C9	1.368 (5)

N2—C13	1.444 (5)	C9—C10	1.390 (5)
N2—C14	1.467 (5)	C9—H9	0.9300
N3—C15	1.312 (6)	C10—C11	1.366 (5)
N3—C17	1.424 (7)	C11—H11	0.9300
N3—C16	1.447 (7)	C12—H12	0.9300
O1—C1	1.277 (4)	C13—H13A	0.9600
O2—C1	1.233 (4)	C13—H13B	0.9600
O3—C4	1.202 (5)	C13—H13C	0.9600
O4—C4	1.316 (5)	C14—H14A	0.9600
O4—H4	0.8200	C14—H14B	0.9600
O5—C7	1.291 (4)	C14—H14C	0.9600
O6—C12	1.252 (4)	C15—H15	0.9300
O7—C15	1.190 (7)	C16—H16A	0.9600
O8—H18	0.8502	C16—H16B	0.9600
O8—H19	0.8500	C16—H16C	0.9600
O9—H20	0.8501	C17—H17A	0.9600
O9—H21	0.8500	C17—H17B	0.9600
C1—C2	1.540 (6)	C17—H17C	0.9600
C2—C3	1.509 (5)		
O5—Cu1—N1	93.51 (12)	C11—C6—C7	120.6 (3)
O5—Cu1—O1	171.55 (12)	C11—C6—C5	117.6 (3)
N1—Cu1—O1	84.86 (12)	C7—C6—C5	121.8 (3)
O5—Cu1—O6	90.85 (11)	O5—C7—C8	119.6 (3)
N1—Cu1—O6	174.78 (12)	O5—C7—C6	124.6 (3)
O1—Cu1—O6	90.42 (11)	C8—C7—C6	115.8 (4)
O5—Cu1—O9	99.55 (11)	C9—C8—C7	123.6 (4)
N1—Cu1—O9	85.39 (11)	C9—C8—Br1	119.4 (3)
O1—Cu1—O9	88.60 (11)	C7—C8—Br1	117.0 (3)
O6—Cu1—O9	96.73 (11)	C8—C9—C10	118.6 (4)
C5—N1—C2	121.4 (3)	C8—C9—H9	120.7
C5—N1—Cu1	126.6 (3)	C10—C9—H9	120.7
C2—N1—Cu1	111.5 (2)	C11—C10—C9	120.9 (4)
C12—N2—C13	121.9 (4)	C11—C10—Br2	120.1 (3)
C12—N2—C14	121.1 (4)	C9—C10—Br2	118.9 (3)
C13—N2—C14	117.0 (3)	C10—C11—C6	120.4 (4)
C15—N3—C17	121.4 (5)	C10—C11—H11	119.8
C15—N3—C16	121.7 (5)	C6—C11—H11	119.8
C17—N3—C16	117.0 (5)	O6—C12—N2	124.5 (4)
C1—O1—Cu1	115.0 (3)	O6—C12—H12	117.8
C4—O4—H4	109.5	N2—C12—H12	117.8
C7—O5—Cu1	128.1 (2)	N2—C13—H13A	109.5
C12—O6—Cu1	121.9 (3)	N2—C13—H13B	109.5
H18—O8—H19	106.6	H13A—C13—H13B	109.5
Cu1—O9—H20	114.0	N2—C13—H13C	109.5
Cu1—O9—H21	98.3	H13A—C13—H13C	109.5
H20—O9—H21	107.2	H13B—C13—H13C	109.5
O2—C1—O1	124.7 (4)	N2—C14—H14A	109.5
O2—C1—C2	118.8 (3)	N2—C14—H14B	109.5
O1—C1—C2	116.2 (3)	H14A—C14—H14B	109.5

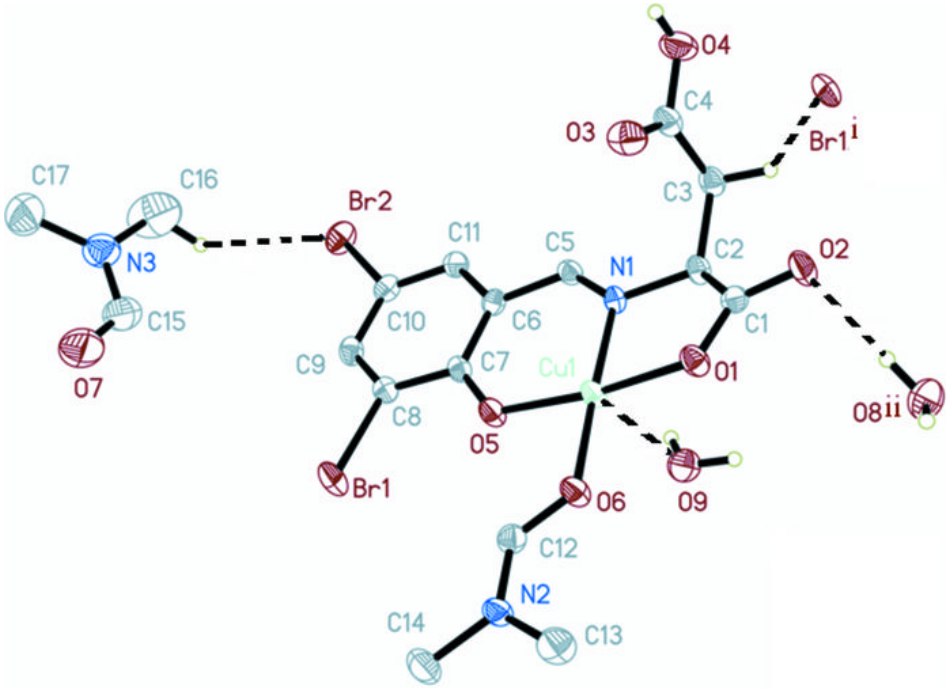
N1—C2—C3	116.8 (3)	N2—C14—H14C	109.5
N1—C2—C1	108.4 (3)	H14A—C14—H14C	109.5
C3—C2—C1	113.9 (3)	H14B—C14—H14C	109.5
N1—C2—H2	105.6	O7—C15—N3	125.9 (6)
C3—C2—H2	105.6	O7—C15—H15	117.1
C1—C2—H2	105.6	N3—C15—H15	117.1
C4—C3—C2	115.5 (3)	N3—C16—H16A	109.5
C4—C3—H3A	108.4	N3—C16—H16B	109.5
C2—C3—H3A	108.4	H16A—C16—H16B	109.5
C4—C3—H3B	108.4	N3—C16—H16C	109.5
C2—C3—H3B	108.4	H16A—C16—H16C	109.5
H3A—C3—H3B	107.5	H16B—C16—H16C	109.5
O3—C4—O4	123.6 (4)	N3—C17—H17A	109.5
O3—C4—C3	125.2 (4)	N3—C17—H17B	109.5
O4—C4—C3	111.3 (4)	H17A—C17—H17B	109.5
N1—C5—C6	125.1 (3)	N3—C17—H17C	109.5
N1—C5—H5	117.4	H17A—C17—H17C	109.5
C6—C5—H5	117.4	H17B—C17—H17C	109.5
O5—Cu1—N1—C5	−0.2 (3)	C1—C2—C3—C4	−65.5 (4)
O1—Cu1—N1—C5	−171.9 (3)	C2—C3—C4—O3	5.9 (6)
O6—Cu1—N1—C5	−146.7 (12)	C2—C3—C4—O4	−175.0 (4)
O9—Cu1—N1—C5	99.1 (3)	C2—N1—C5—C6	174.1 (3)
O5—Cu1—N1—C2	−171.9 (2)	Cu1—N1—C5—C6	3.2 (5)
O1—Cu1—N1—C2	16.4 (2)	N1—C5—C6—C11	177.3 (4)
O6—Cu1—N1—C2	41.6 (14)	N1—C5—C6—C7	−3.0 (6)
O9—Cu1—N1—C2	−72.6 (2)	Cu1—O5—C7—C8	−175.5 (3)
O5—Cu1—O1—C1	−86.2 (8)	Cu1—O5—C7—C6	4.6 (5)
N1—Cu1—O1—C1	−7.0 (3)	C11—C6—C7—O5	178.6 (3)
O6—Cu1—O1—C1	175.2 (3)	C5—C6—C7—O5	−1.0 (6)
O9—Cu1—O1—C1	78.5 (3)	C11—C6—C7—C8	−1.3 (5)
N1—Cu1—O5—C7	−3.6 (3)	C5—C6—C7—C8	179.1 (3)
O1—Cu1—O5—C7	74.9 (9)	O5—C7—C8—C9	−178.1 (4)
O6—Cu1—O5—C7	173.5 (3)	C6—C7—C8—C9	1.8 (5)
O9—Cu1—O5—C7	−89.5 (3)	O5—C7—C8—Br1	2.8 (5)
O5—Cu1—O6—C12	4.6 (3)	C6—C7—C8—Br1	−177.3 (3)
N1—Cu1—O6—C12	151.2 (13)	C7—C8—C9—C10	−2.2 (6)
O1—Cu1—O6—C12	176.2 (3)	Br1—C8—C9—C10	176.9 (3)
O9—Cu1—O6—C12	−95.1 (3)	C8—C9—C10—C11	2.1 (6)
Cu1—O1—C1—O2	−179.4 (3)	C8—C9—C10—Br2	−178.2 (3)
Cu1—O1—C1—C2	−4.2 (4)	C9—C10—C11—C6	−1.7 (6)
C5—N1—C2—C3	36.1 (5)	Br2—C10—C11—C6	178.6 (3)
Cu1—N1—C2—C3	−151.8 (3)	C7—C6—C11—C10	1.3 (6)
C5—N1—C2—C1	166.3 (3)	C5—C6—C11—C10	−179.1 (4)
Cu1—N1—C2—C1	−21.6 (3)	Cu1—O6—C12—N2	175.0 (3)
O2—C1—C2—N1	−167.5 (3)	C13—N2—C12—O6	3.3 (6)
O1—C1—C2—N1	17.0 (4)	C14—N2—C12—O6	−176.1 (4)
O2—C1—C2—C3	−35.7 (5)	C17—N3—C15—O7	−2.2 (10)
O1—C1—C2—C3	148.8 (3)	C16—N3—C15—O7	176.9 (6)
N1—C2—C3—C4	62.1 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O7^{i}	0.82	1.76	2.565 (5)	169
O8—H18···O2^{ii}	0.85	1.93	2.780 (4)	176
O8—H19···O8^{iii}	0.85	2.04	2.889 (3)	175
O9—H20···O8^{iv}	0.85	2.06	2.904 (4)	170
O9—H21···O1^{v}	0.85	2.28	3.116 (4)	167
C3—H3A···Br1^{vi}	0.97	3.13	3.605 (2)	112

Symmetry codes: (i) *x*, −*y*+1, *z*−1/2; (ii) *x*, −*y*+1, *z*+1/2; (iii) −*x*+3/2, *y*−1/2, −*z*+3/2; (iv) −*x*+3/2, −*y*+1/2, −*z*+1; (v) *x*, *y*−1, *z*; (vi) *x*, −*y*, *z*−1/2.

Fig. 1


O4
C4
O3
Br1i
C3
C17
C16
Br2
C11
C5
N1
C2
O2
N3
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C6
C1
C15
C9
C7
Cu1
O1
O7
C8
O5
O8ii
Br1
O6
O9
C12
N2
C14
C13

Fig. 2

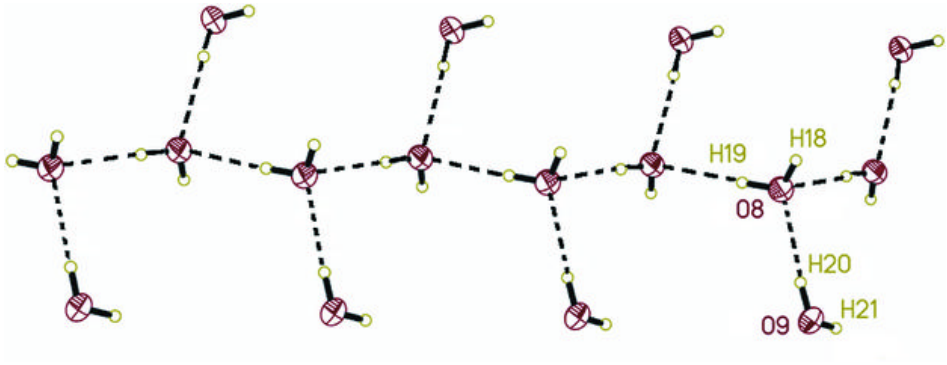

Fig. 3